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Nondestructive Multielement Instrumental Neutron Activation Analysis

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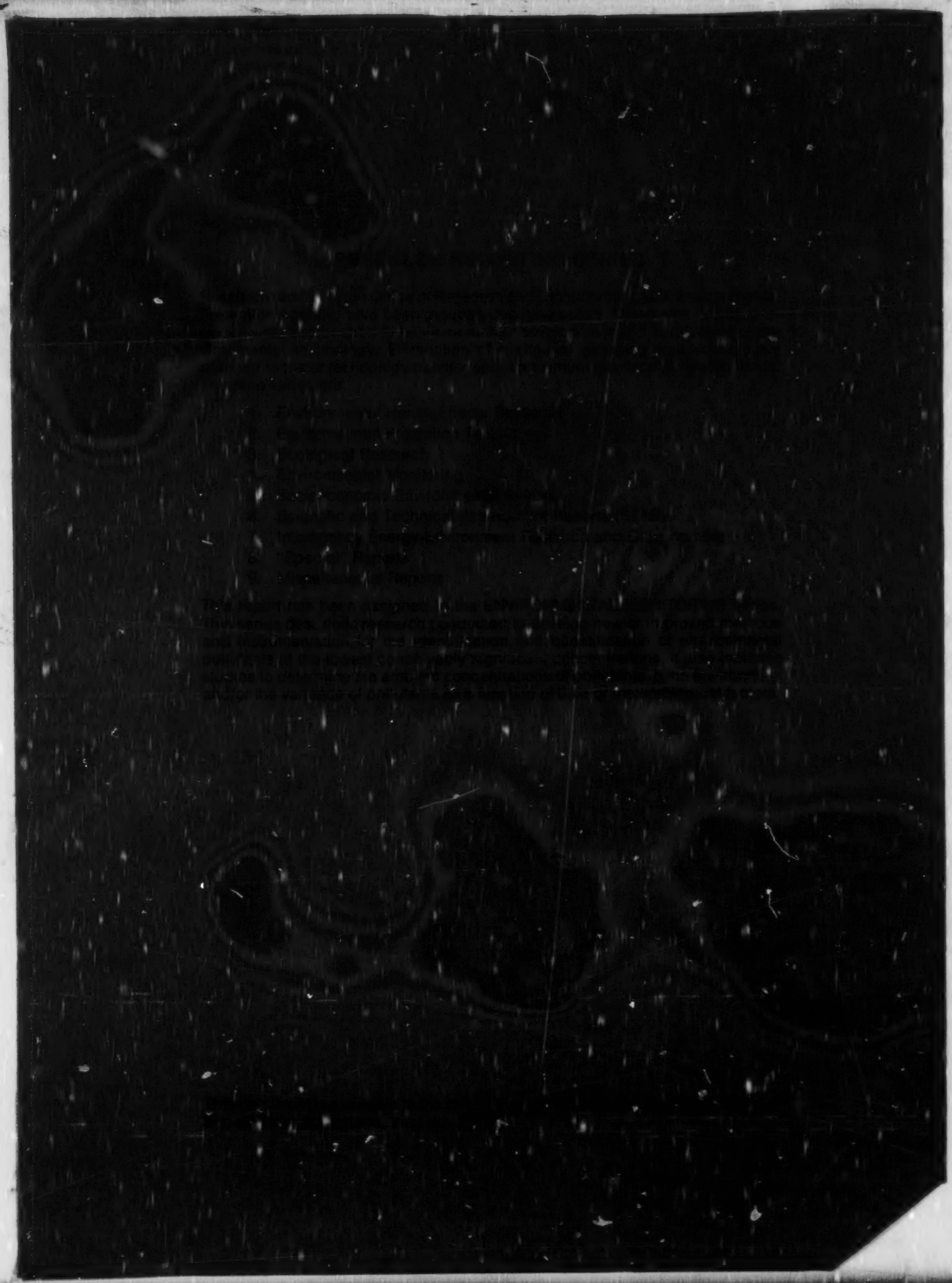

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NONDESTRUCTIVE MULTIELEMENT INSTRUMENTAL NEUTRON ACTIVATION ANALYSIS

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FOREWORD

Nearly every phase of environmental protection depends on a capability to identify and measure chemical pollutants in the environment. As part of this Laboratory's research on the occurrence, movement, transformation, impact, and control of specific environmental contaminants, the Analytical Chemistry Branch develops techniques for identifying and measuring chemical pollutants in water and soil.

This report describes an analytical technique, instrumental neutron activation analysis, for the simultaneous determination of most elements in a wide variety of samples--water, soil, sediments, and biological tissues. It is particularly valuable as a referee method for assessing the accuracy of other more rapid or less expensive procedures. It is sensitive, nearly free of interferences, and avoids pretreatment or concentration of the samples.

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ABSTRACT

A nondestructive instrumental neutron activation analysis procedure permitted accurate and sensitive measurement of most elements with atomic numbers between 11 and 92. The sensitivity of the procedure was dependent on each element's intrinsic characteristics and the sample matrix. Arsenic was used both as an elemental single comparator and as a thermal neutron flux monitor. Comparison conditions were established for both long and short irradiations. Other elemental standards, or unknown samples, were irradiated with flux monitors. Gamma counts of the sample were compared with those of the standards, both having been adjusted to the standard conditions through the flux monitors. The procedure permitted wide latitude in irradiation time, decay time, multichannel analysis time, relative detector-to-sample geometry, and sample size. Analysis of standard reference materials showed that 16 out of 23 elements in Orchard Leaves, for which comparison data were available, agreed within 20%. In Coal 24 out of 30 elemental analyses, and in Coal Fly Ash 21 out of 29 elemental analyses, agreed within 20% of comparison data. Differences greater than 50% were found for antimony, zinc, and a few trace elements near their detection limits.

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SECTION I

CONCLUSIONS

The instrumental neutron activation analysis procedure described here is accurate and sensitive for most elements with atomic numbers between 11 and 92 in most common samples, including water, air, soil and biological materials. It allows a wide latitude in irradiation times, decay times, multichannel analysis time, detector-to-sample geometry, and sample size. In addition, the relatively simple analysis procedure helps to prevent loss or addition of elements, which can occur when more elaborate handling methods are used. The procedure also permits the immediate analysis of standard solutions after preparation, thus minimizing errors caused by element adsorption onto containers. Computers can be used to handle the raw multichannel analysis data.

SECTION II

INTRODUCTION

Environmental pollution control depends on accurate, sensitive analytical procedures to measure a wide range of elements and compounds. Common sample materials, such as water, soil, air, vegetation, and food, may contain many elements at greatly different concentrations. During analysis, elements may be added or lost if the method used requires preparation techniques such as evaporation, ashing, chemical separation, or grinding. Instrumental neutron activation analysis (INAA), however, does not require these techniques and can detect almost any element with atomic numbers between 11 and 92. Any element that, when irradiated with thermal neutrons, produces a radioisotope that in turn produces gamma photons during decay can be detected. The limit of detection for any element is a function of its physical properties, its matrix, and the quality of the instrumentation.

Quantitative nondestructive INAA involves intrinsic properties of each isotope, efficiency of the gamma photon detector, absolute geometry of sample-to-detector position, and sample size. The intrinsic properties of many elements are imprecise, unknown, or erroneous, and neutron fluxes can vary even under ideal conditions.

These problems usually cause the analyst to use the comparative INAA method. In this method, the sample is irradiated along with standards of the elements in question, and elemental composition is calculated by comparison of appropriate gamma photon peak areas. For best results, standards should be used in the same concentration range as unknowns. This method is especially good for analyzing just a few elements but not for analyzing large numbers of elements in widely varying concentrations.

The modified comparative INAA method presented here is practical and useful for analyzing environmental samples of widely differing elemental compositions, and is an accurate referee procedure for more conventional methods.

The common analytical concept of using a set of comparison conditions was the basis of this INAA procedure. Experimental conditions were tailored to fit the particular sample being

analyzed, and data were converted to comparison conditions. Elemental constituents were determined by comparison of sample data to standard data that had been converted to the same comparison conditions. Standards and flux monitors were irradiated simultaneously. To provide valid counting statistics, each standard was measured at a concentration at least two orders of magnitude greater than the sensitivity limit with a count rate to give about 5% actual deadtime of the analog-to-digital converter. Standard sample data were converted to comparison conditions for entry into a catalogue of standard values. Unknown samples were irradiated along with their flux monitors, and counts were converted to comparison conditions for concentration calculations. Because the same flux monitor was used for both standards and unknowns, errors were compensatory.

This use of the flux monitor is a single comparator isotope method (1), in which the nuclear properties of all the other standards isotopes are related empirically to those of the flux monitor.

SECTION III

EXPERIMENTAL

FLUX MONITOR

The flux monitors were arsenic solutions with concentrations of 5.001×10^{-5}g and 5.001×10^{-7} As/g solution in 1.5M HNO_3. Flux values for the 10^{-7} solution were multipled by 100 to fit comparison condition criteria. The solutions have been stable during the year the flux material has been stored in glass.

STANDARDS

Serial dilutions of concentrated standard solutions provided the dilute solutions desired. Usually, three solutions of concentrations differing by two orders of magnitude were prepared; the most dilute was near the detection limit. If possible, each solution was prepared in 1.5M HNO_3 to prevent loss of element to the vessel wall during storage. Standards prepared in only 0.1M HNO_3 will lose elements to the vessel wall. To reduce adsorption errors, standard solutions were used as soon as possible after preparation.

SOLUTION CONTAINERS

Before use, polyethylene sample vials were soaked for several hours in 1.0N NaOH, rinsed carefully, and dried for several hours at 65°C. This treatment apparently converts metallic ions in the plastic to refractory oxides that are insoluble and do not contaminate samples. A HNO cleaning technique (2) appears to enhance sample contamination with elements from the polyethylene container.

SAMPLES

National Bureau of Standards standard reference materials (SRM), were used to test this procedure. They were SRM 1571, Orchard Leaves; SRM 1632, Trace Elements in Coal; and SRM 1633, Trace Elements in Coal Fly Ash. Neutron irradiations and multichannel analyses were tailored to fit the materials. Standards were used as received; wet to dry ratios were obtained with nonirradiated samples. For each SRM, six replicates were analyzed using long irradiations, and six replicates using short irradiations.

IRRADIATIONS

All irradiations were made in the Georgia Institute of Technology Research Reactor, which has a high ratio of thermal neutrons to fast neutrons (325:1 for cobalt and 69:1 for aluminum). Each sample and flux monitor was placed in a pre-washed, laboratory-grade polyethylene, 1.5-ml (2/5-dram) vial, which was heat sealed and further encapsulated in a polyethylene bag.

For short irradiations (≤ 1 hr.), three vials were encapsulated together in a polyethylene bag and irradiated simultaneously in a polyethylene "rabbit" container designed for use with the pneumatic tube system. The rabbit's dimensions allowed the sample to be placed between two flux monitors along the longitudinal axis of the rabbit, where 5% flux variations have been measured. Neutron flux at the sample, which was the average of that measured by the two flux monitors, was about 1×10^{13} neutrons/cm^2/sec. Length of irradiation was adapted to the sample. After irradiation, samples and flux monitors were transferred to tared vials for weighing, and gamma photon emissions were counted using multichannel analysis. To minimize geometry differences, flux monitor and sample volumes were kept as similar as practicable.

Long irradiations (usually about 8 hr.) were made in vertical thimbles positioned in the reactor core to give a flux of about 2×10^{13} neutrons/cm^2/sec. Two flux monitors and two samples were placed in a square configuration with flux monitors and samples at alternate corners of the square. The group of four was then encapsulated in polyethylene, and ten groups were placed in an aluminum can to be inserted into the reactor. After irradiation and after short-lived constituents had died out (usually about four days), flux monitors and samples were transferred to tared polyethylene vials, weighed, and counted. For each sample, average counts for two sample duplicates and two flux monitors were used for calculations.

Quartz encapsulation must be used for samples in which mercury and iodine are to be measured, because these elements are lost from polyethylene vials. Other elements in samples encapsulated in quartz can be compared accurately with standards measured in plastic, when flux monitors are similarly treated, except for the elements that are impurities in quartz.

MULTICHANNEL ANALYSIS

Multichannel analysis counts were made using a Nuclear Data, Inc., ND4420 system, which includes an ND812 computer with 32K of memory, a low-density disc, and software. The analog-to-digital converter has a 8192 channel resolution and a 100 MHz digitizing rate; data were collected on 4096 channels.

A lithium-drifted germanium (Ge(Li)) solid state detector was used to detect gamma photons. It was a closed-end, modified-coaxial type. When compared to a 3 x 3 in. sodium iodide scintillation detector, for Co^{60}, the Ge(Li) detector had an efficiency of 11.5%; a full width, half maximum (FWHM) resolution of 1.95 keV; and a peak-to-Compton ratio of 37:1. The detector was protected from extraneous radiation by a vault with 10-cm thick lead walls lined with cadmium and copper sheets. A plastic sample holder was attached to the detector for reproducible vertical and horizontal sample positioning. Solid samples that did not fill their vials were rotated during counting to minimize geometry errors. The ND4420 system was controlled through a teletype, and data were output through either the teletype or a high-speed printer.

Samples and flux monitors were positioned in front of the detector so that the deadtime of the ADC was less than 10%, usually less than 7%, and a counting time appropriate to the sample was used. Samples subjected to short irradiations were counted immediately after irradiation and again after the decay of very short-lived elements. Appropriate counting times were used to obtain the best practical statistics for detected isotopes. Samples subjected to long irradiations were counted as soon as possible, usually after about four days to obtain data on relatively long-lived isotopes, and again several days later to measure very long-lived isotopes. In the latter case, counting times of 2 hr. to 16 hr. were often needed.

CALCULATIONS

During multichannel analysis, each detected gamma emission was processed through a preamplifier, a linear amplifier, and an analog-to-digital converter, and was stored in a memory bank in a few channels. The mean of these channels was proportional to the energy of that peak. This process produced a spectrum of peaks. Collection and processing of a gamma spectrum for each sample was controlled by a Nuclear Data proprietory peak search computer program (ND1087-04 disc-based nuclide identification program) which found each peak (resolving any doublets), determined its energy, calculated its gross area, determined the background count, and corrected the gross area to give the net area of the peak. Using the energies determined, the program then performed a "nuclide identification" routine using a catalogue of isotopes furnished by the analyst, who then programmed the computer to determine concentrations.

Two isotope catalogues were compiled, one for short-lived isotopes and one for long-lived isotopes. Each catalogue entry contained the isotope's symbol, half-life, gamma energy, gamma abundance, and the factor to be used to calculate its concentration. When possible, three abundant gammas that were free from interference were entered for each element. Other

gammas and induced X-rays were also entered to help verify the element's presence.

Comparison conditions were established for short and long irradiations, based on the 5.001×10^{-5} g As/g solution standard. Short irradiation conditions required a 10-min. irradiation and flux monitor count rate of 1000 counts/g/sec. at time zero, which was defined as the time at the end of irradiation. For long irradiations, comparison conditions required an 8-hr. irradiation and flux monitor count rate of 100,000 counts/g/sec. at time zero.

The peak area was a function of the quantity of the isotope present, the live time of counting, the decay time between the end of irradiation and the beginning of multichannel analysis, the time of irradiation, the neutron flux, and the sample position with respect to the detector. To obtain concentration data, sample peak areas must be compared to those obtained with standards. For comparison, peak areas must be adjusted to time zero and must reflect equivalent irradiation times and flux count rates.

$$R = \frac{A \times e^{\lambda(Td + Tc)}}{Tl \times W \times Rx} \qquad (1)$$

where R = count rate, detected gammas/sec/gram, at time zero

A = measured area (counted gammas)

λ = isotope activity coefficient - ln 2/half life of isotope

Td = decay time from time zero to beginning of multichannel analysis

Tc = that portion of elapsed counting time Te that must be added to Td to give the true decay time

Tl = live counting time

W = weight of sample

Rx = dry/wet ratio (a correction for moisture content of solid samples)

Some calculations are necessary to determine Tc, which must be added to Td to produce the true time that produced the observed count rate. The average count rate, A/Tl, occurs at a time during counting that is not necessarily at Te/2, where Te is the elapsed counting time. When the isotope half life, Th, is long relative to Te, Tc is not significantly different from Te/2. When Te/Th is less than 0.01, Tc = Te/2.

However, when Te/Th is greater than 0.01, then the variation from Te/2 becomes significant (3).

$$Tc = -1/\lambda \times \ln[1/\lambda\ Te \times (1-e^{-\lambda\ Te})] \qquad (2)$$

When the experimental irradiation time Ti differs from the comparison condition time, the count rate, R, must be adjusted by an irradiation factor, I.

$$I = \frac{(1-e^{-\lambda To})}{(1-e^{-\lambda Ti})} \qquad (3)$$

where Ti = irradiation time

To = comparison irradiation time

The count rate adjusted for the irradiation time is given by S = RI.

If S_s is the adjusted count rate for the standard, and S_f for its flux monitor, the standard count rate, C_o, at the comparison flux count rate, F_o, (1000 or 100,000, depending on standard solution concentration) is:

$$C_o = \frac{S_s \times F_o}{S_f} \qquad (4)$$

Actual concentrations were calculated by comparing unknowns to standards. A concentration factor, C_f, was calculated for each gamma used by the equation Cf = Cs/Co, where Cs = concentration of element that produced the isotope measured. These concentration factors were included in the isotope catalogues stored in the computer.

To determine standard concentration factors, solutions of three concentrations differing by two orders of magnitude were analyzed. Six determinations were made at each concentration. The regression coefficient of peak area against concentration did not differ significantly from the slope determined using only the most concentrated standard solution; therefore, the concentration factors used were those calculated for the most concentrated standard solution.

Letting Su be the adjusted count rate for each gamma detected in an unknown, and S_f for its flux monitor, the unknown concentration Cu can be obtained from:

$$Cu = \frac{Su \times Cf \times Fo}{Sf} \qquad (5)$$

SECTION V

RESULTS AND DISCUSSION

The need to detect and measure the widest possible range of contamination has extended conventional methodology to new levels of sensitivity and created a need for referee methods to ensure that the extensions were valid. For INAA to meet this need in elemental analysis, it had to be able to analyze almost all elements in all sample types. The procedure described here fulfills this need to a large degree.

Arsenic was chosen as a flux monitor because it has many favorable characteristics for this type of work. Arsenic trioxide is a primary analytical standard that is stable in solution. If acidified to 1.5M with HNO_3, the solution does not change concentration when stored in good quality glass and can be used for both long and short irradiations. Measurable emissions can be obtained after only 3 sec. of irradiation; flux monitors irradiated for 8 hr. can be counted after 4 or 5 days. When activated, its primary gamma emission is at 559.1 keV. Its low intensity gamma at 562.8 does not interfere if a good resolution detector is used and if the computer can compute the background. Because the primary gamma emission of so many elements is between 100 keV and 1000 keV, the arsenic gamma at 559.1 keV is an acceptable compromise. At that energy, detector efficiencies are still quite high as compared to maximum efficiencies near 100 keV.

This method allows the simultaneous analysis of most elements with atomic numbers between 11 and 92 in a single sample, even in complex samples, such as soils. This precludes having to make, irradiate, and count a number of multielement standard solutions or filter paper standards on which a number of elements have been deposited.

To optimize results, the analyst must be able to control as many variables as possible. With this procedure, some control is possible over the sample size, the encapsulation material, the irradiation time, the decay time, the counting time, the number of times the sample is counted, and the sample positioning with respect to the detector. The analyst must also ensure that the sample and flux monitor receive the same treatment. Both must be encapsulated similarly, have about the same volume, be irradiated simultaneously, and be counted with the same

positioning with respect to the detector. Because counts have to be adjusted to comparison conditions, flux monitor decay and counting times may be varied to optimize flux monitor counting requirements.

Using a flux monitor makes the procedure independent of a number of factors that the analyst cannot control. The reactor used in this study operates on a daily cycle and never attains a state of flux equilibrium. Samples are subjected to flux variations as xenon burnup occurs, control rod positions are changed, and temperatures equilibrate.

The analyst has little control over the positioning within the reactor of samples to be irradiated for 8 hr. and turntables are not available to equalize the flux exposure of these samples. Reactor scrams or shutdowns also introduce irradiation variations, and changes in fuel rod positioning or replacement of rods may occur during a research project. Because flux monitors are irradiated and counted simultaneously with unknowns, this procedure is independent of neutron flux variations.

As long as both sample and its flux monitor are treated alike, analyses are not affected by changes in detectors, amplifiers, and ADC's. During this investigation, two amplifiers and two ADC's were used. The detector had to be remounted, which changed the position of the detector crystal within its own housing.

Because the flux monitor method allows the standards to be measured independently of the samples, dilute solutions of standards can be run as soon as they are made up, thus reducing the effect of adsorption on the container.

The flux monitor, however, has not eliminated all INAA problems. When a sample matrix has a high level of some elements, the usefulness of the INAA procedure can be reduced appreciably. For example, a long irradiation of large quantities of phosphorus produces so much ^{32}P, which decays and produces a very energetic beta, that the resultant bremsstrahlung masks most gamma emissions with energies less than 500 keV. Some isotopes produce gammas with nearly equal energies; when one is dominant, the other is lost. Magnesium (843.8 keV gamma) and manganese (846.7 keV) are examples; both produce secondary gamma emissions that permit analytical determination. As detectors improve, this problem will be reduced.

Another problem is sample size. The National Bureau of Standards certificates for the Coal and Coal Fly Ash reference materials indicated homogeneity within ±5% if 250-mg samples were used. In this study, sample weights were generally 40 to 200 mg. This was required because the level of induced radiation was so great that a compromise had to be made between

sample size, radiation intensity (deadtime to the detector), and geometry of sample to detector. Only a flexible procedure allows such a trade off. The deadtime of the ADC is measurable, but the deadtime of the amplifier is not known or measurable at this time. The deadtime of the ADC had to be kept low to ensure insignificant amplifier deadtime and to prevent gain shifting, which makes it impossible to match found energy peaks with catalogue values.

Elements for which standards were not available were not reported. Mercury and iodine were not reported because of significant losses from the polyethylene vials. In general, the values for elements measured in standard reference materials agree with those reported by others (Tables 1-3). In the orchard leaves, sample data for 23 elements can be compared to published values. Of these, 16 agree within 20% and 19 within 50%. Of the 30 elements in coal for which comparison data are available, 24 are within 20% and 29 within 50%. In coal fly ash, there are 29 elements to compare, 21 being within 20% and 27 within 50%. Our results for antimony do not compare well with other data, possibly because of sample inhomogeneity, as has been suggested by Ondov et al. (5). Zinc analyses in the present work agree well with the reference value for orchard leaves but are significantly low for coal and coal fly ash. No explanation is readily available. Other elements for which poor agreement was found were present at near the detection limits.

This single comparator method of INAA is effective because of the improved Ge(Li) detectors available today as compared with those in use as recently as 1973-74 (7). These detectors, in conjunction with fast analog-to-digital converters and computers, make this procedure practical, versatile, and accurate.

State-of-the-art detectors, with their high peak-to-Compton ratios, give the analyst assurance that the back-scatter of high energy gamma photons will not mask the peaks of lower energy photons. Even in the presence of a 0.3% sodium concentration, for example, other isotopes are readily analyzed. The improved resolution of these detectors means two gamma photon peaks that could interfere with one another may be resolved. For example, magnesium and manganese, 843.8 keV and 847.6 keV, respectively, may be measured in each others presence. Generally, the doublet peak resolution routine of the computer aids in such analyses.

The high ratio of thermal-to-fast neutrons, furnished by the Georgia Institute of Technology research reactor, meant that interference resulting from fast and epithermal neutron reactions was minimal. Because each standard can be measured separately, however, these reactions can be measured and the necessary corrections entered into the computer to be applied when necessary.

Table 1. ELEMENTS DETECTED IN ORCHARD LEAVES STANDARD REFERENCE MATERIAL[a]

Element	Concentration, µg/g[b]		
	This Work	Morrison & Potter (4)	NBS
Al	450 ± 120	440	-
As	9.8 ± 0.2	10	14 ± 2
Au	0.0025 ± 0.0006	.001	-
Ba	50 ± 6	51	-
Br	14 ± 1	8.25	10[c]
Ca	2.0 ± 0.1%	2.095%	2.09 ± 0.03%
Cl	890 ± 70	790	700[c]
Co	0.16 ± 0.03	0.1	0.2[c]
Cr	2.0 ± 0.2	2.5	2.3[c]
Cu	-	10	10 ± 1
Dy	0.15 ± 0.07	-	-
Eu	0.024 ± 0.003	0.3	-
Fe	307 ± 40	290	300 ± 20
K	1.6 ± 0.05%	1.505%	1.47 ± 0.03%
La	1.5 ± 0.06	1.2	-
Mg	0.67 ± 0.04%	.595%	0.62 ± 0.02%
Mn	98 ± 5	86	91 ± 4
Na	98 ± 21	77	82 ± 6
Nd	18 ± 15	-	-
Rb	12 ± 0.7	11	12 ± 1
Sb	6.8 ± 0.7	3	-
Sc	0.071 ± 0.007	0.205	-
Se	-	0.08	0.08 ± 0.01
Sm	0.12 ± 0.04	0.145	-
Sr	41 ± 14	40	37[c]
Th	0.70 ± 0.014	-	-
Ti	34 ± 7	-	-
U	-	-	0.029 ± 0.005
V	0.76 ± 0.10	<0.65	-
Zn	27 ± 4	25	25 ± 3

[a]National Bureau of Standards SRM 1571.

[b]Values in µg/g unless % indicated.

[c]Information values only.

Table 2. ELEMENTS DETECTED IN COAL STANDARD REFERENCE MATERIAL[a]

Element	Concentration, μg/g[b]			
	This Work	Ondov et al. (5)	Klein (6)	NBS
Al	1.8 ± 0.1%	1.85 ± 0.13%	1.90%	-
As	5.6 ± 0.4	6.5 ± 1.4	5.5	5.9 ± 0.6
Au	0.0023 ± 0.0011	-	-	-
Ba	330 ± 36	352 ± 30	405	-
Br	23 ± 1	19.3 ± 1.9	14.2	-
Ca	0.36 ± 0.12%	0.43 ± 0.05%	0.44%	-
Cl	1100 ± 100	890 ± 125	1000	-
Co	5.4 ± 0.5	5.7 ± 0.4	5.9	6[c]
Cr	16 ± 1	19.7 ± 0.9	21 ± 2	20.2 ± 0.5
Cs	1.8 ± 0.2	1.4 ± 0.1	1.4	-
Cu	-	-	18[d]	18 ± 2
Dy	1.8 ± 0.2	-	-	-
Eu	0.39 ± 0.05	0.33 ± 0.04	0.21	-
Fe	0.94 ± 0.05%	0.84 ± 0.04%	0.84%	0.87 ± 0.03%
K	0.28 ± 0.01%	0.28 ± 0.03%	0.290%	-
La	12 ± 0.8	10.7 ± 1.2	10.5	-
Mg	0.11 ± 0.02%	0.20 ± 0.05%	0.248%	-
Mn	50 ± 5	43 ± 4	46 ± 3	40 ± 3
Mo	4.7 ± 2.0	-	3.4	-
Na	420 ± 45	414 ± 20	390	-
Nd	48 ± 6	-	-	-
Rb	20 ± 4	21 ± 2	19.5	-
Sb	5.9 ± 4	3.9 ± 1.3	4.45	-
Sc	3.9 ± 0.3	3.7 ± 0.3	4.5	-
Se	3.3 ± 0.4	3.4 ± 0.2	3.05	2.9 ± 0.3
Sm	1.7 ± 0.1	1.7 ± 0.1	-	-
Sr	170 ± 12	161 ± 16	123	-
Ta	0.33 ± 0.12	0.24 ± 0.04	0.17	-
Th	3.2 ± 0.2	3.2 ± 0.2	3.0	3.60[c]
Ti	940 ± 87	1100 ± 100	930	800[c]
U	-	-	1.26	1.4 ± 0.1
V	44 ± 3	36 ± 3	40 ± 3	35 ± 3
W	0.70 ± 0.28	0.75 ± 0.17	-	-
Yb	0.76 ± 0.07	0.7 ± 0.1	-	-
Zn	18 ± 6	30 ± 10	34[d]	37 ± 4

[a]National Bureau of Standards SRM 1632.

[b]Values in μg/g unless % indicated.

[c]Information values only.

[d]Values determined with X-ray fluorescence.

Table 3. ELEMENTS DETECTED IN COAL FLY ASH STANDARD REFERENCE MATERIAL[a]

Element	Concentration, μg/g[b]			
	This Work	Ondov et al. (5)	Klein (6)	NBS
Al	13 ± 1%	12.7 ± 0.5%	12.5%	-
As	52 ± 3	58 ± 4	54	61 ± 6
Au	0.032 ± 0.003	-	-	-
Ba	2800 ± 350	2700 ± 200	2780	-
Br	7.7 ± 0.5	12 ± 4	6.0	-
Ca	4.9 ± 0.1%	4.7 ± 0.6%	4.34%	-
Cl	-	42 ± 10	-	-
Co	37 ± 4	41.5 ± 1.2	46	38[c]
Cr	100 ± 16	127 ± 6	138	131 ± 2
Cs	10 ± 1	8.6 ± 1.1	-	-
Cu	-	-	133[d]	128 ± 5
Dy	160 ± 58	-	-	-
Eu	2.9 ± 0.5	2.5 ± 0.4	2.86	-
Fe	6.9 ± 0.8%	6.2 ± 0.3%	6.37%	-
K	1.7 ± 0.7%	1.61 ± 0.15%	1.8%	1.72[c]
La	100 ± 8	82 ± 2	82	-
Mg	1.3 ± 0.2%	1.8 ± 0.4%	1.98%	-
Mn	540 ± 38	496 ± 19	460	493 ± 7
Mo	57 ± 24	-	-	-
Na	2300 ± 200	3200 ± 400	3070	-
Nd	330 ± 39	-	-	-
Rb	110 ± 14	125 ± 10	120[d]	-
Sb	17 ± 2	6.9 ± 0.6	7.8	-
Sc	29 ± 4	27 ± 1	32	-
Se	8.3 ± 0.7	10.2 ± 1.4	9.35 ± 0.03[e]	9.4 ± 0.5
Sm	12 ± 2	12.4 ± 0.9	15	-
Sr	1700 ± 56	1700 ± 300	1301[d]	1380[c]
Ta	2.1 ± 0.2	1.8 ± 0.3	1.6	-
Th	25 ± 3	24.8 ± 2.2	26	24[c]
Ti	7500 ± 300	7400 ± 300	6420	-
U	26 ± 15	12.0 ± 0.5	11.8	11.6 ± 0.2
V	310 ± 28	235 ± 13	240	214 ± 8
W	4.2 ± 0.7	4.6 ± 1.6	-	-
Yb	5.7 ± 0.7	7 ± 3	-	-
Zn	120 ± 40	216 ± 25	208[d]	210 ± 20

[a]National Bureau of Standards SRM 1633.

[b]Values in μg/g unless % indicated..

[c]Information values only.

[d]Values determined with X-ray fluorescence.

[e]Values determined with gas chromatography with microwave emission spectrometric detection.

This single comparator INAA may be established wherever there are enough thermal neutrons available along with state-of-the-art detectors coupled to suitable computers, including dedicated minicomputers. Results are dependent on the standards analyzed plus a wide range of variables over which the analyst has control.

SECTION VI

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